Organic Matter and Water Stability of Field Aggregates Affected by Tillage in South Dakota

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Tillage has been associated with soil organic matter (SOM) decline. A case study of two adjacent farms was conducted in eastern South Dakota. One farm used no-till (NT) and the other used chisel tillage (CT). We hypothesized that soil under NT, compared with tillage, would have both greater quantity and greater quality of SOM and that this improved SOM condition would result in increased water stable aggregation (WSA). A rotary sieve was used to sort dry field aggregates into six size groups: <0.4, 0.4 to 0.8, 0.8 to 2.0, 2.0 to 6.0, 6.0 to 19.0, and >19 mm. Water stable aggregation, soil organic C (SOC), N, glomalin, and basidiomycete fungi were measured. Fine particulate soil organic matter (fPOM, 0.5–0.053 mm) and coarse particulate organic matter (2.0–0.5 mm) were isolated by sieving. Quantitative solid-state ^{13}C nuclear magnetic resonance was used to determine C type in humic acid, humin, and whole soil. The fPOM/SOM ratio was greatest in <0.4-mm aggregates and 24% greater in NT than CT. Soil organic C was greatest in 0.8- to 2.0-mm aggregates and 11% greater in NT than CT. Average WSA was 63% greater under NT than under CT. Aggregate wettability was less under NT than CT. Slower water uptake under NT might be attributed to a greater abundance of wax-type C under NT than under CT. We conclude that NT, compared with CT, resulted in better SOM quality during the course of 10 yr. Improved SOM quality was related to improved WSA.

Abbreviations: AM, arbuscular mycorrhizal; cPOM, coarse particulate organic matter; CT, chisel tillage; DPMAS, direct-polarization magic-angle spinning; EC, electrical conductivity; fPOM, fine particulate organic matter; HA, humic acid; IRTG, immunoreactive total glomalin; NMR, nuclear magnetic resonance; NT, no-till; POM, particulate organic matter; SOC, soil organic carbon; SOM, soil organic matter; TPOM, total particulate organic matter; WSA, water stable aggregation.

The quantity and quality of SOM provides an important link between management and soil function. It is generally accepted that conversion to crop production practices has caused a decline in SOM compared with the original grassland levels throughout the Great Plains (Campbell and Souster, 1982; Monreal and Janzen, 1993; Allmaras et al., 2000). Tillage has caused soil C losses from 28 to 77% depending on geographic location (climate) and soil type (Paustian et al., 1997). Changes in agricultural management from CT to NT and enhancement of rotation complexity have increased the accumulation of SOC (West and Post, 2002).

Conservation of SOM is essential to sustaining crop productivity (Doran et al., 1998) and maintaining the soil resource because SOM is strongly linked to soil fertility and desirable soil tilth (Carter, 2002). Boyle et al. (1989), in a review of the influence of SOM on soil aggregation and water infiltration, concluded that SOM had a disproportionate effect

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on soil physical behavior. Water stability of soil aggregates has been shown to be dependent on the quantity and quality of organic materials (Tisdall and Oades, 1982). Bruce et al. (1992) determined that increased phytomass input to a loamy sand increased aggregate stability and water infiltration. On long-term tillage, residue management, and N-fertility plots, Pikul and Zuzel (1994) found that an increase in SOM increased the porosity of surface crusts in a silt loam soil.

The effect of management on soil aggregation is not always clearly defined, nor is the time necessary to effect change readily understood. Degens (1997) provided a review of the function of labile organic bonding and binding agents related to soil aggregation. Degens (1997) suggested that conclusions on soil stabilization resulting from controlled incubation studies contribute little to understanding biological processes under field conditions.

Fungi play an important role in the formation and stabilization of soil aggregates through the adhesive effects of metabolic products and entanglement of soil particles. Exudates from basidiomycete fungi are important in soil aggregation, but short lived as binding agents (Caesar-TonThat and Cochran, 2000). Arbuscular mycorrhizal (AM) fungi produce an exudate called glomalin, which is a stable, Fe-containing glycoprotein (Wright and Upadhyaya, 1996) thought to be important in soil aggregation (Wright et al., 1999). Recently, Gadkar and Rillig (2006), cloned the gene for glomalin production from AM fungal hyphae and provided the first evidence for the identity of the glomalin protein in the AM fungi *Glomus*. In a recent review, Treseder and Turner (2007) concluded that soil glomalin stocks were positively correlated with net primary productivity but not with AM fungi abundance. Glomalin concentrations in soil were often greater, however, in the presence of AM host plants that maintained relatively high AM fungi colonization rates.

New microaggregates are thought to form around decomposing pieces of root-derived particulate organic matter (POM) inside macroaggregates (Gale et al., 2000a). Particulate organic matter is physically defined as the organic material isolated in the fraction 0.053 to 2.00 mm. It is an intermediate between fresh plant litter and humified SOM, and has been shown to be more sensitive to changes in management than total SOM (Cambardella et al., 2001). In undisturbed soils, POM is derived primarily from roots (Gale et al., 2000a,b). Six et al. (2000) proposed an aggregate "life cycle," suggesting that fine intraaggregate POM is formed as it becomes encrusted with clay particles and microbial products within macroaggregates. As macroaggregates degrade, stable microaggregates become the nuclei for the formation of new macroaggregates. Nichols and Wright (2006) showed that POM contained significant amounts of glomalin.

Humification of SOM under different agricultural systems results in unique chemical constituents of humic materials that may improve soil aggregate stability. Ding et al. (2002) showed that the composition of humic acid (HA) under conventional tillage was less aliphatic (alkyl C) and more aromatic than HA developed under conservation tillage. Investigations of SOM decomposition using quantitative solid-state ^{13}C nuclear magnetic resonance (NMR) have shown that aliphatic structures are more recalcitrant and increase in abundance relative to other fractions as decomposition proceeds (Ussiri and Johnson, 2003). The increase in the proportion of aliphatic materials may be the result of utilization of easily decomposed carbohydrates (O-alkyl C) by microorganisms. Ussiri and Johnson (2003) suggested using the ratio of alkyl C to O-alkyl C (R) as an index to the degree of decomposition. Gregorich et al. (2001) found that the composition of crop residue returned to the soil had little effect on the chemical composition of SOM as determined by ^{13}C NMR. Stearman et al. (1989) found that on a Loring silt loam (a fine-silty, mixed, active, thermic Oxyaquic Fragiudalf), HA under no-till (treatments having greater amounts of C) had greater aliphatic to aromatic ratios and suggested that this characteristic might be due to earlier stages of decomposition.

Water stable soil aggregation has been related to differences in aggregate wettability, and soil wettability is related to the chemical constituents of the SOM. Chenu et al. (2000) found that SOM associated with clay minerals increased hydrophobicity, resulting in resistance of aggregates to slaking. Shepherd et al. (2001) concluded that greater aggregate stability of soil under pasture was due to the presence of a protective water-repellent lattice of long-chain polymethylene compounds around soil aggregates. Cultivation of prairie soils of central South Dakota has resulted in a decline in wettability compared with never-tilled grassland (Eynard et al., 2004a).

There is a scarcity of studies dealing with the properties of naturally formed field aggregates, their distribution, and their breakdown. Frequently, work on soil aggregate stability starts with aggregates well under 2 mm in size, and many investigators use wet-sieving techniques (Kemper and Rosenau, 1986) as a means to isolate small aggregate units (Franzluebbers et al., 2000; Shaver et al., 2002; Mikha and Rice, 2004; Olchin et al., 2008). Thus, only aggregates that are stable in water are examined further, which leads to the question of whether measured aggregate properties are largely an artifact of the chosen method of separation. The method of fractionation has been shown to influence physical, chemical, and biological properties of different sized soil aggregates (Ashman et al., 2003; Sainju, 2006). We believe that it is important to test natural field aggregates because they serve is an indicator of the capability of surface soil to resist breakdown and sealing (Ben-Hur and Lado, 2008). We hypothesized that soil under no-till, compared with tillage, would have both greater quantity and better quality of SOM and that this improved SOM condition would result in increased WSA. Our objectives were to measure the WSA of naturally formed soil aggregates from two tillage systems and elucidate the relationships between selected properties of the SOM and WSA.

MATERIALS AND METHODS

Field Site and Soil Sampling

A case study was conducted from 8 Nov. 2001 to 23 June 2004 on adjacent farms in eastern South Dakota (Fig. 1). This period of time covered both the corn (*Zea mays* L.) and soybean [*Glycine max* (L.) Merr.] crops, but for brevity only data from the 2002 soybean year are reported here. One farm chiseled and disked fields each fall (CT), and the other farm used NT. On the NT farm, primary tillage was last used in 1992. Under CT, corn and soybean were also row cultivated each year. Primary tillage under CT was approximately 160 mm deep. Farms were located about 24 km southeast of Brookings, SD (44°14.5′ N, 96°41.2′ W). The crop rotation

on each farm was corn–soybean and both farms were in the same crop phase of the rotation each year. Farms were planted to corn in 2001 and soybean in 2002.

On each farm, four plots (pseudoreplications) were established at near-summit slope positions within the same soil mapping unit (Fig. 1). Plots on the NT farm were approximately 150 m from plots on the CT farm and each plot was 30 m long by 30 m wide.

The soil is a Vienna–Brookings silty clay loam complex on a 0.6% slope (Brookings series: fine-silty, mixed, superactive, frigid Pachic Hapludolls; Vienna series: fine-loamy, mixed, superactive, frigid Calcic Hapludolls). Depth to free $CaCO_3$ is approximately 50 cm. Both the Vienna and Brookings soil series consist of very deep, well to moderately well drained soils formed in loess over glacial till.

To characterize the general soil profile of each farm, triplicate soil cores 32 mm in diameter and 80 mm in length were taken from each plot to a depth of 240 mm in June 2002. Soil bulk density (ρ_b), SOC, total N, pH, and electrical conductivity (EC) were determined on these samples. Soil organic C and total N were determined by combustion using a LECO CN 2000 analyzer (LECO Corp., St Joseph, MI). All samples were ground and passed through a 0.5-mm sieve before analysis. Soil pH and EC were measured using a 1:1 soil/water mixture. Soil texture was measured on samples collected from the top 50 mm.

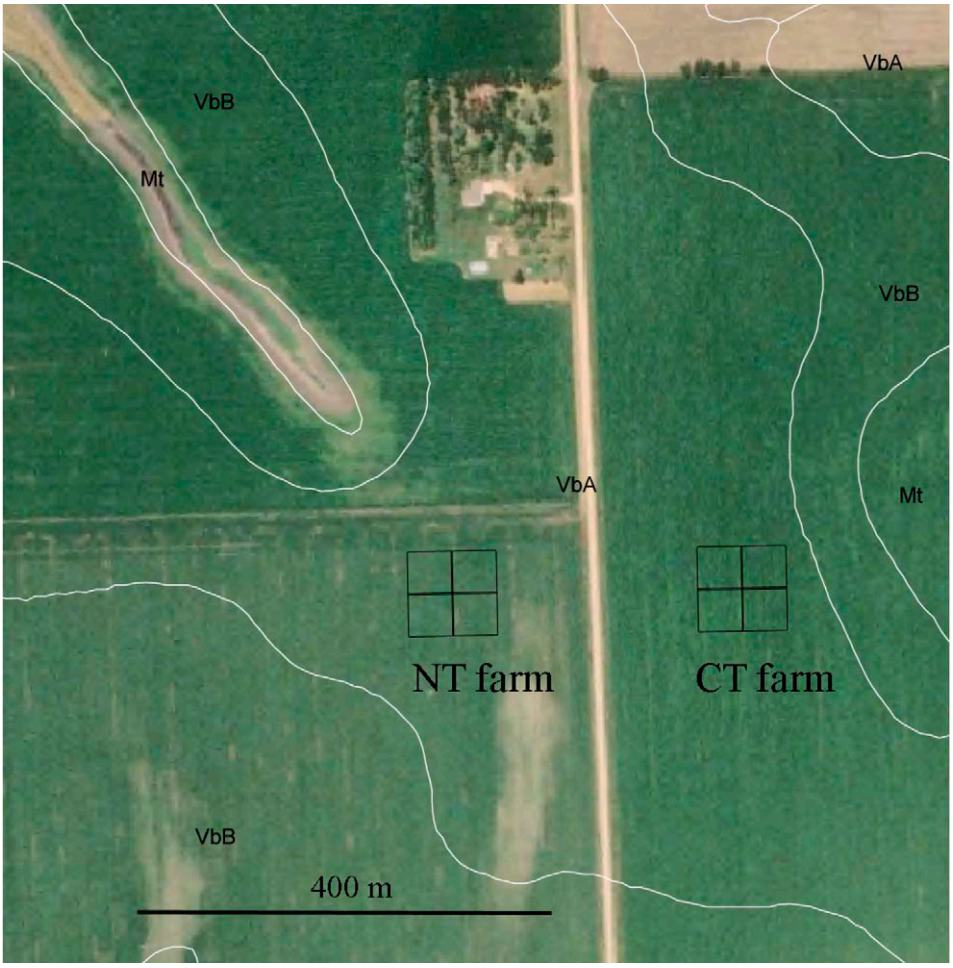

Fig. 1. Soil map (Schaefer, 2004) showing location of farms (Township 109 N, Range 48 W, Sections 16 and 17) using no-till (NT) and chisel tillage (CT). The rectangular grid identifies location of the four plots (pseudoreplications) on each farm (actual slope 0.6%). Soil is a Vienna–Brookings complex (VbA, 0–2% slope).

We used a rotary sieve (Chepil, 1962) to sort naturally formed field aggregates into six size groups. About 10 kg of soil was collected with a scoop shovel from the surface 50 mm on 13 June and 1 Oct. 2002. On each sampling date, we randomly collected six subsamples within each plot to acquire 10 kg of soil. After collection, the samples were spread in thin layers on greenhouse benches to air dry. Samples were not crushed. The rotary sieve uses a controlled rate of feed to convey a bulk sample into a nest of rotating sieves (operating at 10 rpm). Samples were sorted into six size groups: <0.4, 0.4 to 0.8, 0.8 to 2, 2 to 6, 6 to 19, and >19 mm. This range of sieve sizes is also common to those rotary sieves used in Kansas and North Dakota (Zobeck et al., 2003).

Measurements of WSA, SOM, POM, and SOC were made on aggregates collected on 13 June and 1 Oct. 2002. Measurements of glomalin, basidiomycete fungi, HA, and C type (alkyl C, O-alkyl C, aromatic C, and carboxyl C) were made on aggregates collected on 13 June 2002.

Measurements

Water Stable Aggregation

Water stability of aggregates collected in June and October 2002 was measured using a sieve and sieving procedure as described by Kemper and Rosenau (1986). The screen size (sieve opening) on our apparatus was 0.71 mm. Tests were conducted on about 5 g of dry aggregates from size groups 0.4 to 0.8, 0.8 to 2, 2 to 6, and 6 to 19 mm. Because of the large size of aggregates in the 6- to 19-mm size group, we selected about 5 g of aggregates that were approximately 10 mm in size and these aggregates were placed on the screen without crowding. Duplicate measurements were made on all aggregates. Water stable aggregation was expressed as the percentage of soil remaining on the sieve after 5 min relative to the initial mass of soil used for the test. Stability calculations were corrected for sand content by subtracting the mass of sand remaining on the sieve from the initial soil mass and the mass of soil remaining on the sieve after 5 min.

Wettability

The wetting rate of the soil under a water potential of –0.98 kPa was measured on aggregates of approximately 10-mm diameter from each plot (Quirk and Panabokke, 1962). Briefly, individual soil aggregates were placed on Büchner funnels having sintered glass plates, the water potential was adjusted to –0.98 kPa (achieved using a hanging water column of 100 mm measured from the top of the sintered glass plate), and the rate of water uptake by the aggregates was measured by the movement of the meniscus in a graduated capillary tube. Total porosity of aggregates was calculated from aggregate density (ρ_{agg}) measured by the clod method (Blake and Hartge, 1986) using a particle density of 2.65 Mg m^{-3}.

Particulate Soil Organic Matter

Particulate organic matter was measured by dispersing and sieving, using the weight loss-on-ignition (LOI) method of Cambardella et al. (2001) and further described by Gajda et al. (2001). We used an ignition temperature of 450°C for 4 h and 30- and 10-g samples for POM and SOM, respectively, as recommended by Cambardella et al. (2001) and also used by other investigators (Gajda et al., 2001; Mikha et al., 2006). Particulate organic matter remaining on the 0.5-mm sieve was termed coarse POM (cPOM) and had a size range of 0.5 to 2.0 mm; POM retained on the 0.053-mm sieve was called fine POM (fPOM) and had a size range of 0.053 to 0.5 mm. The mass of POM was determined by LOI.

Soil organic matter was measured using LOI (Cambardella et al., 2001; Gajda et al., 2001). Fine POM and cPOM were expressed relative to SOM (fPOM/SOM and cPOM/SOM ratios) for each aggregate group.

Soil Carbon

Soil C for aggregate groups <0.4, 0.4 to 0.8, 0.8 to 2, 2 to 6, 6 to 19, and >19 mm was measured by combustion using a LECO CN 2000 analyzer. All samples were ground and passed through a 0.5-mm sieve before analysis. Before grinding, visible pieces of crop residue were removed. For noncalcareous soils (Nelson and Sommers, 1982), soil C can be considered to be SOC. Generally, the pH values for calcareous soils are within a range of 7.5 to 8.5 (Loeppert and Suarez, 1996). The pH of our soil profile samples (Table 1) indicated that pretreatment to remove carbonates (Nelson and Sommers, 1982) before dry combustion was not necessary. Soil C will therefore be referred to as SOC.

Basidiomycetes

The quantity of basidiomycetes in aggregate size groups 0.8 to 2, 2 to 6, and 6 to 19 mm was determined using an enzyme-linked immunosorbent assay (ELISA) protocol as described in Caesar-TonThat et al. (2001). Absorbance (Abs450) was read at a dual wavelength of 450/655 nm using a BioRad 550 microplate reader controlled by a computer using the Plate Reader Manage program (BioRad, Hercules, CA). All incubation steps on aggregate material were performed at room temperature. All samples were processed in triplicate. Two independent tests were made on each aggregate sample. Measurements were made on whole aggregates before wet sieving and on the soil retained on the sieve following the wet sieve test.

Glomalin

Glomalin was extracted from the soil of each aggregate group using the extraction procedure of Wright et al. (1999) except that 1 g of sample was extracted by using 8 mL of extraction solution. Extraction was performed using 50 mmol L^{-1} sodium citrate, pH 8.0. This extraction protocol may coextract other soil proteins, and the extract has been renamed *glomalin-related soil protein* (Rillig, 2004), however, we will refer to glomalin-related soil protein as *glomalin*. All extractions were performed at 121°C for 1 h, and the extraction cycles were repeated seven times. The supernatant from each extraction cycle was combined and assayed for glomalin concentration using an ELISA protocol and the antiglomalin monoclonal antibody as described by Wright and Upadhyaya (1998). This fraction will be called *immunoreactive total glomalin* (IRTG).

Glomalin was also extracted from five fractions of SOM in the 2- to 6-mm aggregate size group. Fractions were: (i) visible plant residue picked from the soil before wet sieving; (ii) fPOM from water stable aggregates; (iii) cPOM from water stable aggregates; (iv) fPOM from aggregates that slaked during wet sieving; and (v) cPOM from aggregates that slaked during wet sieving. Two glomalin fractions (citrate-extractable and recalcitrant glomalin [Nichols and Wright, 2005, 2006; Wright et al., 2006]) were extracted from 0.4- to 4-g samples of the five SOM fractions.

Citrate-extractable glomalin was extracted with 50 mmol L^{-1} sodium citrate at pH 8.0. The recalcitrant glomalin fraction was extracted with 100 mmol L^{-1} sodium pyrophosphate at pH 9.0 (Wright et al., 2006). Repeated extractions (until the solution was straw colored) were performed at 121°C for 1 h. The supernatant from each extraction cycle was combined and assayed for glomalin concentration. Each extractant (citrate or pyrophosphate) was analyzed separately. Glomalin concentrations in SOM materials were measured using a Bradford total protein assay (Wright and Upadhyaya, 1996).

Humic Acid

Soil was fractionated into humin and HA following Stevenson (1994). Thirty grams of soil from aggregate size groups <0.4, 0.4 to 0.8, 0.8 to 2, 2 to 6, and 6 to 19 mm (collected on 13 June 2002) were extracted for HA. Humic acid was separated from the humin and the inorganic fraction using repeated extractions with 0.5 mol L^{-1} NaOH under N_2. Humin and HA fractions were dried, ground, and analyzed for total C, N, and organic C type.

Solid-State Carbon-13 Nuclear Magnetic Resonance

Organic C type in the HA, humin, and soil was determined by quantitative ^{13}C NMR. Samples of HA and humin consisted of bulked samples (replications and aggregate size groups <0.4, 0.4 to 0.8, 0.8 to 2, 2 to 6, and 6 to 19 mm). Soil was a bulked sample consisting of 10-mm aggregates from four replications. All samples were ground and passed through a 0.5-mm sieve. The quantitative ^{13}C NMR technique, described by Mao et al. (2000), utilizes direct-polarization magic-angle spinning (DPMAS) at high rotation speeds (13 kHz) combined with a T_1^C correction obtained from cross-polarization total sideband suppression (CP/T_1–TOSS) experiment. The recycle delays used for DPMAS were determined for each sample. The number of scans recorded ranged between 5000 and 35,000. All samples, except HA fractions, were treated with dilute solutions of HF to remove Fe before NMR analysis (Keeler and Maciel, 2003). Samples were packed in a 4-mm-diameter zirconia rotor with a Kel-F cap. Spectra were acquired at 75 MHz on a Bruker ASX300 spectrometer. The ^{13}C NMR spectra were integrated according to the following chemical shift regions: 0 to 50 ppm, alkyl C (aliphatic C); 50 to 108 ppm, O-alkyl C (carbohydrate C); 108 to 162 ppm, aromatic C; and 162 to 212 ppm, carboxyl C. The distribution among these four major C types was calculated by integration using software supplied with the spectrometer operating system.

Statistics

Statistical comparisons of measured soil properties were made using one-way and two-way analysis of variance (Minitab Release 14, Minitab Inc., State College, PA). Fixed effects were tillage (CT and NT). Time of sampling (13 June and 1 Oct. 2002) and replications were treated as random effects in the combined analysis. Treatment effects (one-way ANOVA) were considered significant for $P \leq 0.05$. The effect of time and tillage (two-way ANOVA) and the time × tillage interaction were evaluated using a general linear model.

Stepwise regression was used to identify significant predictors of WSA for aggregates of size groups 0.8 to 2.0 and 2.0 to 6.0 mm. Measurements on these size groups included fPOM, cPOM, total particulate organic matter (TPOM), C and N of HA, SOC, N, basidiomycete fungi, IRTG, and SOM. We had too few measurements of the distribution of organic C types with ^{13}C DPMAS NMR for regression modeling.

RESULTS AND DISCUSSION

Site Characteristics

There were no differences between farms in soil texture in the top 50 mm. The average clay content was 385 g kg^{-1} under NT (standard error = 8.66) and 375 g kg^{-1} under CT (standard error = 5.0). The average sand content was 212 g kg^{-1} under NT (standard error = 7.5) and 217 g kg^{-1} under CT (standard error = 2.5). Under NT, ρ_b for soil depths of 0 to 80, 80 to 160, and 160 to 240 mm was significantly greater at each depth than under CT (Table 1). For the 240-mm depth profile, ρ_b was 10% greater under NT than under CT.

The soil C distribution was stratified with depth such that NT had a greater (but not significant on a mass basis) SOC concentration in the top 80 mm than CT (Table 1). On a volumetric basis, there was 15% more SOC within the top 80 mm under NT than CT, and 8% more SOC in the top 240 mm under NT than CT. The C/N ratio in the top 80 mm was greater under NT than CT, probably reflecting the accumulation of residue at the surface. At the 80- to 160-mm depth, the SOC concentration was greater under CT than NT. These depth distributions of C are consistent with differences in residue placement between tillage treatments. In the case of NT, all residues remained on the surface. Under CT, an increase of SOC concentration at the 80- to 160-mm depth suggests residue placement (Allmaras et al., 1988; Staricka et al., 1991) at this depth. There were no differences in soil pH or EC between the treatments (Table 1).

Table 1. Soil properties at depths of 0 to 80, 80 to 160, and 160 to 240 mm under no-till (NT) and chisel tillage (CT) treatments†.

Soil property and tillage system	0–80 mm	80–160 mm	160–240 mm	Mean
Bulk density, g cm^{-3}				
NT	1.18	1.39	1.46	1.34
CT	1.05	1.24	1.34	1.21
P value	0.028	0.002	0.002	0.019
Carbon, g kg^{-1}				
NT	30.2	23.7	22.9	25.6
CT	29.4	26.2	23.3	26.3
P value	NS‡	0.005	NS	NS
C/N ratio				
NT	10.8	10.5	10.5	10.6
CT	10.5	10.2	10.2	10.3
P value	0.028	NS	NS	0.004
pH				
NT	6.6	6.4	6.8	6.6
CT	6.3	6.2	6.6	6.4
P value	NS	NS	NS	NS
Electrical conductivity, dS m^{-1}				
NT	0.396	0.295	0.308	0.333
CT	0.360	0.306	0.316	0.327
P value	NS	NS	NS	NS

† Measurements made on soil cores collected on 13 June 2002.

‡ Not significant at $P \leq 0.05$.

Soil Aggregate Properties

Water-Stable Aggregation

Aggregates in the size range of 0.4 to10 mm under NT were more stable in water compared with those under CT. The average WSA was 38% under NT compared with 23% under CT. Table 2 shows the average WSA for soil samples collected on 13 June and 1 Oct. 2002 when both farms were in soybean. During the course of the study (four sampling dates from 8 Nov. 2001 to 23 June 2004 covering both corn and soybean crops), WSA remained significantly greater ($P = 0.001$, $n = 128$) under NT (31%) than CT (19%). Water stability of aggregates increased with aggregate size on both the NT and CT farms (Table 2). It is probable that larger aggregates simply disintegrate at a slower rate than smaller aggregates.

Our findings are comparable to that of Eynard et al. (2004b). They found similar significant differences in WSA of 1- to 2-mm sized aggregates (32% WSA under NT compared with 25% WSA under CT) in a study of Ustolls from six locations in central South Dakota.

Carbon and Nitrogen

Aggregates from NT had significantly greater SOC in all aggregate groups compared with aggregates from CT (Table 3). On both NT and CT, the greatest concentration of SOC was found in aggregate sizes 0.8 to 2.0 mm. The average concentration of SOC in all size groups was 32.3 g kg^{-1} under NT, compared with 29.6 g kg^{-1} under CT. Table 3 shows the average SOC for soil samples collected on 13 June and 1 Oct. 2002 when both farms were in soybean.

Table 2. Water-stable aggregation (WSA) of aggregates sized from <0.4 to >19.0 mm collected from the top 50 mm of no-till (NT) and chisel tillage (CT) treatments†.

Tillage treatment	WSA						
	<0.4 mm	0.4–0.8 mm	0.8–2.0 mm	2.0–6.0 mm	10.0 mm	>19.0 mm	Mean
	%						
NT	NM‡	18.5	26.9	35.6	71.9	NM	38.2
CT	NM	11.8	15.5	18.7	47.5	NM	23.4
	P values (WSA)						
Tillage		0.001	0.001	0.001	0.001		0.005
Date		NS§	NS	NS	0.001		NS
Date × tillage		0.025	NS	NS	NS		NS

† Measurements of WSA made on soil collected on 13 June and 1 Oct. 2002.

‡ Not measured.

§ Not significant at $P \leq 0.05$.

Table 3. Soil organic C (SOC), C/N ratio, ratio of fine particulate organic matter to soil organic matter (fPOM/SOM), and SOM of six soil aggregate groups sized from <0.4 to >19.0 mm from the top 50 mm of no-till (NT) and chisel tillage (CT) treatments†.

Property and tillage system	<0.4 mm	0.4–0.8 mm	0.8–2.0 mm	2.0–6.0 mm	6.0–19.0 mm	>19.0 mm
SOC, g kg^{-1} soil						
NT	32.4	32.8	34.4	33.2	31.0	30.1
CT	29.5	29.7	31.0	30.2	28.8	28.4
P value						
Tillage	0.010	0.001	0.007	0.001	0.007	0.017
Date	NS‡	NS	NS	0.003	NS	NS
Tillage × date	0.044	NS	NS	NS	NS	NS
C/N						
NT	10.6	10.7	10.8	10.7	10.6	10.6
CT	10.2	10.1	10.3	10.4	10.1	10.2
P value						
Tillage	0.02	0.001	0.001	0.001	0.004	0.005
Date†	NS	0.020	NS	NS	NS	NS
Tillage × date	NS	NS	NS	NS	NS	NS
fPOM/SOM, %						
NT	20.09	13.26	13.39	16.23	15.11	12.35
CT	16.15	9.04	9.14	11.47	10.45	9.62
P value	0.022	0.002	0.001	0.001	0.001	0.053
SOM, g kg^{-1} soil						
NT	76.44	74.58	83.80	81.98	76.93	74.62
CT	76.56	75.06	78.10	79.26	71.96	71.46
P value	NS	NS	0.002	NS	0.039	NS

† Soil C and N measured on soil samples collected on 13 June and 1 Oct. 2002; fPOM/SOM ratios measured on samples collected on 1 Oct. 2002.

‡ Not significant at $P \leq 0.05$.

The C/N ratio was significantly greater under NT than CT for all aggregate groups (Table 3). The average C/N ratio of aggregates was 10.7 under NT and 10.2 under CT. Under NT, the greatest C/N ratio was found in aggregate sizes 0.8 to 2.0 mm, whereas under CT, the greatest C/N ratio was found in aggregate sizes 2.0 to 6.0 mm (Table 3). There were no differences in the C/N ratios of aggregates due to time of sampling (13 June or 1 Oct. 2002) except within aggregate sizes 0.4 to 0.8 mm.

Particulate Organic Matter

Particulate organic matter has been shown to be a labile fraction of SOM (Paul et al., 2001; Cambardella and Elliott, 1992; Cambardella et al., 2001). We show POM as a fraction of SOM (Table 3) and also as a fraction of soil mass (Table 4) for each aggregate group. Average fPOM, expressed as a fraction of SOM (Table 3), was significantly greater under NT than CT within all aggregate groups. We found similar results when fPOM was expressed as a fraction of soil mass (Table 4). The greatest concentration of fPOM was within aggregates <0.4 mm (Tables 3 and 4). Additionally, fPOM, whether expressed as a fraction of the soil (Table 4) or a fraction of the SOM (Table 3), comprised a greater fraction of TPOM (Table 4) than did cPOM for both NT and CT. A greater concentration of POM in the undisturbed NT soil may be a consequence of an accumulation of root materials and less organic matter decomposition (Gale et al., 2000a,b). The average SOM of aggregates (Table 3) was significantly greater in size groups of 0.8 to 2.0 and 6.0 to 19.0 mm under NT than CT. For both SOC and SOM, the greatest concentration was found in midsized aggregates (Table 3). That was not the case, however, for the distribution of fPOM, where the greatest concentration was in the smallest size groups (Tables 3 and 4).

Glomalin, Basidiomycetes, and Humic Acid

Within aggregate groups, there were no significant ($P \leq 0.05$) differences between treatments in IRTG, but the concentration of IRTG was consistently greater under NT than under CT (Table 5). The immunoreactive component of glomalin in soil is strongly correlated with exudates of AM fungal hyphae (Wright, 2000). We found no significant differences between treatments in the concentration of glomalin (Bradford total protein assay) associated with the POM of water stable aggregates. There was a significant difference, however, in the concentration of glomalin associated with POM of water stable vs. slaked aggregates. For both NT and CT, the average concentration of glomalin was nearly five times greater in POM associated with water stable aggregates than POM associated with slaked aggregates. The greatest concentration of glomalin was 22 g glomalin kg^{-1} material and this was found in the visible plant materials. By comparison, there was an average (treatments and POM fraction) of 9 g glomalin kg^{-1} material in POM.

Table 4. Fine particulate organic matter (fPOM) and total particulate organic matter (TPOM) as a fraction of soil. Aggregates sized from <0.4 to >19.0 mm were collected from the top 50 mm of no tillage (NT) and chisel tillage (CT) treatments†.

Tillage system	<0.4 mm	0.4–0.8 mm	0.8–2.0 mm	2.0–6.0 mm	6.0–19.0 mm	>19.0 mm
	g kg^{-1} soil					
fPOM						
NT	15.36	9.89	11.22	13.33	11.65	9.24
CT	12.44	6.79	7.14	9.10	7.52	6.88
P value	NS‡	0.004	0.001	0.002	0.002	0.049
TPOM						
NT	17.76	17.16	19.64	17.11	13.35	10.52
CT	13.73	10.50	13.35	14.88	8.89	7.59
P value	NS	0.001	0.002	NS	0.004	0.042

† fPOM and TPOM measured on soil samples collected on 1 Oct. 2002.

‡ Not significant at $P \leq 0.05$.

The average number of basidiomycetes in the soil remaining on the screens following wet sieving was significantly greater (40%) under NT than CT (Table 5) for the 0.8- to 2.0-mm aggregates. For both NT and CT, the 0.8- to 2.0-mm aggregates had the greatest absorbance value (number of basidiomycetes). These fungi play an important role in the formation and stabilization of soil aggregates (Caesar-TonThat and Cochran, 2000) through the adhesive effects of microbial metabolic products and entanglement of soil particles by filamentous fungi.

There were small differences in the C/N ratios of HA between NT and CT (Table 5). Generally, aggregates <0.4 mm had the greatest C/N ratios. We think that the difference in C/N ratios among aggregate groups provides evidence that "less humified" materials (having greater C/N ratios) were present within the smaller aggregates. Stevenson (1994) suggested a relation between organic matter decomposition and the C/N ratio of HA such that soil mixing and aeration would encourage decomposition, resulting in a narrowing of the C/N ratio of HA. Chefetz et al. (2002) found that HA of aggregates, containing freshly deposited organic matter, showed greater C/N ratios. The aggregate size evaluated by Chefetz et al. (2002) roughly corresponds to our <0.4-mm aggregates.

Table 5. Carbon/nitrogen ratio of humic acid, immunoreactive total glomalin (IRTG), and basidiomycete assay of six soil aggregate groups sized from <0.4 to >19.0 mm from the top 50 mm of no-till (NT) and chisel tillage (CT) treatments†. The basidiomycete assay (absorbance at 450 nm) was conducted on material retained on the sieve following wet sieving.

Property and tillage system	<0.4 mm	0.4–0.8 mm	0.8–2.0 mm	2.0–6.0 mm	6.0–19.0 mm	>19.0 mm
C/N of humic acid						
NT	11.2	11.0	10.4	10.4	10.5	NM‡
CT	11.0	10.9	10.2	10.3	10.3	NM
P value	NS§	NS	NS	NS	0.006	
IRTG, g kg^{-1}						
NT	0.52	0.55	0.58	0.57	0.54	0.58
CT	0.44	0.48	0.46	0.48	0.46	0.44
P value	NS	NS	NS	NS	NS	NS
Basidiomycete assay						
NT	NM	NM	0.757	0.519	0.474	NM
CT	NM	NM	0.532	0.506	0.429	NM
P value			0.018	NS	NS	

† Measurements made on soil samples collected on 13 June 2002.

‡ Not measured.

§ Not significant at $P \leq 0.05$.

Nuclear Magnectic Resonance Spectra

Quantitative solid-state ^{13}C DPMAS NMR was used to examine the HA and humin fractions as well as the whole soil. In general, all spectra (Fig. 2) showed broad but resolved peaks for carbons in the alkyl (0–50 ppm), O-alkyl (50–110 ppm), aromatic (110–160 ppm), and carboxyl (160–220 ppm) regions of the spectrum.

Results from the integration of ^{13}C NMR spectra of HA, humin, and whole soils are presented in Table 6. Distribution of organic C types in the humin fraction was similar to the whole soil, indicating that humin was representative for the whole mineral soil analyzed. The aromatic C was the most abundant fraction in all samples, followed by O-alkyl C for the whole soil and humin and by carboxyl C for the HA fractions. The relative abundance of carboxyl C in the HA fraction was greater than in humin and whole soils. In addition to greater carboxyl C, HA fractions had lower O-alkyl C (carbohydrate C) content than humin and whole soils.

Interpretation of NMR spectra for soils is done in terms of C type rather than attributing a specific chemical shift to specific compounds (such as cellulose or lignin). An index of the degree of decomposition (*R*), defined as the ratio of alkyl C (aliphatic C) to O-alkyl C (carbohydrate C), was calculated following Ussiri and Johnson (2003). Results show that treatment did not significantly affect the distribution of organic C in HA (Table 6). There were notable differences in *R* values, however, for humin fractions and whole soils by treatment. Higher values of *R* for humin and whole soils under CT indicate an increase in decomposition of SOM under tillage. This finding is consistent with Ghidey and Alberts (1993), who reported faster decomposition of buried material than material on the

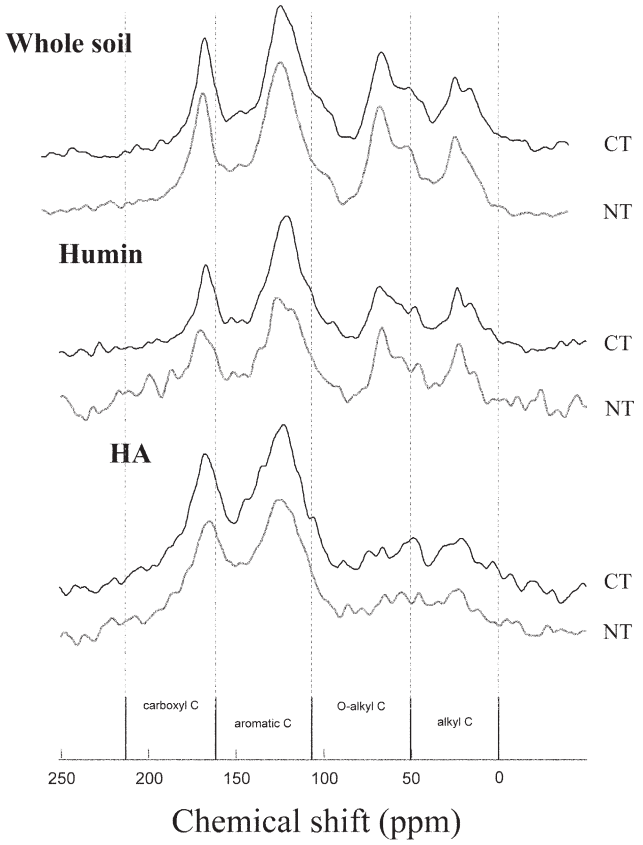

Fig. 2. Solid-state ^{13}C direct-polarization magic-angle spinning nuclear magnetic resonance (DPMAS NMR) spectra for whole soil, humin, and humic acid (HA) fractions for chisel tillage (CT) and no-till (NT) farms. Vertical lines show distribution of four major C types: 0 to 50 ppm, alkyl C (aliphatic C); 50 to 108 ppm, O-alkyl C (carbohydrate C); 108 to 162 ppm, aromatic C; and 162 to 212 ppm, carboxyl C.

Table 6. Distribution of organic C type in humin, humic acids, and whole soil based on solid-state ^{13}C direct-polarization magic-angle spinning nuclear magnetic resonance (DPMAS NMR), from the top 50 mm of no-till (NT) and chisel tillage (CT) treatments†.

Fraction	Tillage	Carbon distribution				
		Alkyl C	O-alkyl C	Aromatic C	Carboxyl C	*R*‡
		%				
Whole soil	CT	19.4	28.1	35.3	17.1	0.69
	NT	15.7	26.6	37.3	20.4	0.58
Humin	CT	17.5	25.1	39.8	17.6	0.69
	NT	14.0	23.6	39.3	23.0	0.59
Humic acid	CT	14.4	19.1	40.3	26.1	0.76
	NT	13.6	18.6	41.5	26.3	0.73

† Measurements were made on soil samples collected on 13 June 2002.

‡ Index of decomposition defined as the ratio between alkyl and O-alkyl C (Ussiri and Johnson, 2003).

surface. Sleutel et al. (2007) compared two adjacent farm fields under reduced tillage and CT and found evidence, using pyrolysis–field ionization mass spectroscopy, of a less decomposed character of SOM under reduced tillage.

Types of water-repellent substances in soil organic matter include aliphatic components and waxes, represented in the quantitative C^{13} NMR spectra by the chemical shift region between 0 and 50 ppm. The region between 0 and 20 ppm of the C^{13} NMR spectra is attributed to methyl C, mainly from branched alkyl carbons. The region between 20 and 33 ppm of the same spectra is attributed to methylene C, mainly polymethylene C from short and long chains due to the presence of waxes. These two regions were integrated and the polymethylene C (20–33 ppm) to methyl C (0– 20 ppm) ratio was calculated. Under NT, the ratio was 2 compared with 1.5 under CT for the whole soil. The difference was more pronounced for the humin samples, where the ratio was 3 under NT compared with 1.5 under CT. No difference occurred for HA.

Wettability

Soil organic matter affects aggregate wettability by decreasing the hydration rate, increasing the hydrophobic surface area, and stabilizing soil pores. Hydrophilic compounds such as polysaccharides can also buffer the soil against water potential fluctuations. These processes have been summarized by Eynard et al. (2006). The initial rate of soil water uptake, shown as the slope of the uptake curve between 5 and 15 min in Fig. 3, was nearly twofold less under NT (0.008 $m^3\ m^{-3}\ min^{-1}$) than under CT (0.013 $m^3\ m^{-3}\ min^{-1}$). At 60 min, there were no differences in water-filled pore space between NT and CT (Fig. 3). The average pore space for NT was 0.40 and for CT 0.49 (Fig. 3).

Differences in wettability may be due to differences in soil C type responsible for water repellency (especially waxes). Hydrophobicity of soils may be related to the aliphatic C present in organic matter (Ellerbrock et al., 2005). We found a greater percentage of total aliphatic C (0–50 ppm) in the soil under CT compared with NT (Table 6, alkyl C), but in the spectral region between 20 and 33 ppm we found a greater percentage of methylene C, mainly polymethylene C from short and long chains due to the presence of waxes, under NT. The greater percentage of polymethylene C under NT might impart a degree of hydrophobicity and help to explain the reduced water uptake under NT.

Aggregates from NT exhibited a slower rate of water uptake compared with CT (Fig. 3) and also greater water stability (Fig. 4). Shepherd et al. (2001) concluded that the high aggregate stability of a humic soil under pasture was due to the presence of a protective water-repellent lattice of long-chain polymethylene compounds around soil aggregates. Eynard et al. (2004a) found a higher abundance of tubular pores under NT than under CT. Tubular pores that are interconnected could serve as conduits to rapidly equalize pore pressure dur-

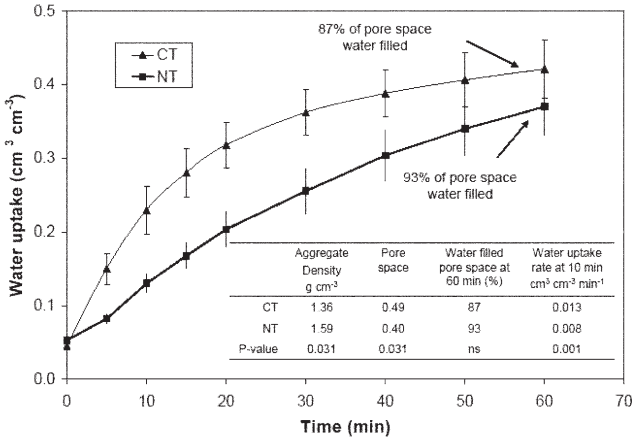

Fig. 3. Water uptake by soil aggregates of approximately 10-mm diameter from no-till (NT) and chisel tillage (CT) farms. Pore space [1 – (aggregate density)/(particle density)] of aggregates was calculated from aggregate density measured by the clod method (Blake and Hartge, 1986) and using a particle density of 2.65 g cm^{-3}. Soil aggregates were under a water potential of –0.98 kPa (100-mm hanging water column).

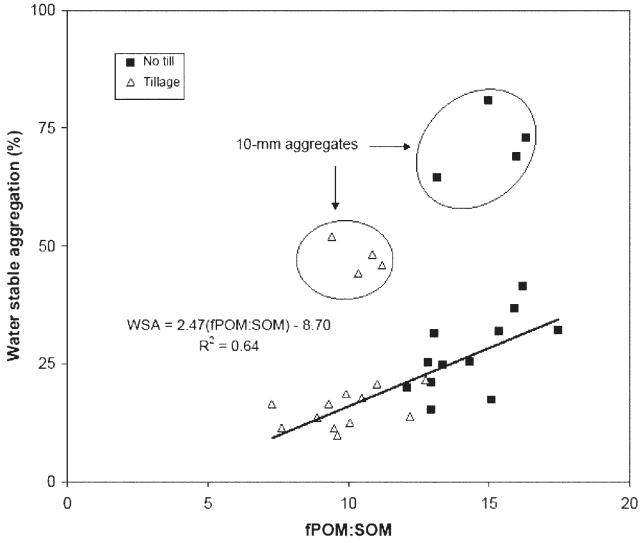

Fig. 4. Relationship of water stable aggregation (WSA) to the fine particulate organic matter/soil organic matter (fPOM/SOM) ratio. Soil aggregates tested were from size groups 0.4 to 0.8, 0.8 to 2.0, and 2.0 to 6.0 mm and 10-mm aggregates from size group 6.0 to 19.0 mm of no-till (NT) and chisel tillage (CT) farms. The linear regression does not include tests on the 10-mm aggregates.

ing water submersion and result in decreased slaking of soil structural units.

Regression Modeling

Linear regression showed that the WSA of aggregates was significantly ($P \leq 0.05$) related to fPOM/SOM ratios ($P < 0.0001$), TPOM ($P < 0.017$), the C/N ratio of HA ($P = 0.005$), SOC ($P = 0.003$), soil N ($P = 0.018$), and SOM ($P = 0.012$). The best single-component predictor of aggregate stability was the fPOM/SOM ratio (Fig. 4). This finding is consistent with Pikul et al. (2007), who also found a significant, positive relationship ($r^2 = 0.79$) between WSA and fPOM/SOM ratios across seven sites under diverse crop rotations and tillage. The best two-component model included the fPOM/SOM ratio and the C/N ratio of HA ($r^2 = 0.86$), providing the following regression equation:

$$\text{WSA} = -201 + 2.11(\text{fPOM/SOM}) + 19.2(\text{C/N of HA})$$

There was little improvement in the prediction of aggregate stability by including additional parameters beyond the fPOM/SOM ratio and the C/N ratio of HA.

CONCLUSIONS

Collectively, our results provide insights into the biological, chemical, and physical changes that occur in soil as a result of NT management. We found that soil under NT had a greater fPOM/SOM ratio than CT, and as the fPOM/SOM ratio increased, WSA increased. The soil environment under NT, compared with CT, is highly conducive to the accumulation of POM, because decaying plant root systems remain undisturbed. Fungi play an important role in the formation and stabilization of soil aggregates through the adhesive effects of metabolic products and entanglement of soil particles. The environment under NT would favor fungal growth, and we found a greater population of fungi under NT than under CT.

Differences in the decomposition of organic materials under these two tillage systems resulted in unique chemical constituents of SOM. There was a greater abundance of C associated with wax compounds (methylene C, mainly polymethylene C from short and long chains due to the presence of waxes) in both the whole soil and humin under NT than CT. Organic compounds may impart some degree of water repellency, thereby improving WSA. Water stable aggregation was 60% greater under NT than under CT. It is probable that the greater concentration of glomalin, fungal populations, and C associated with wax compounds found under NT, compared with CT, contributed to greater WSA under NT than CT. Tillage practices that improve soil aggregate stability also help to maintain surface conditions resistant to erosive forces. We conclude that NT during the course of 10 yr resulted in favorable changes in the quantity and quality of SOM under NT. An outcome of the improvement in SOM under NT was improved WSA.

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